



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE BOARD OF PATENT APPEALS AND INTERFERENCES

In re application of

Confirmation No. 6935

Tatsuhiko SHIBUYA et al.

: Docket No. 00774/98-129 TOK/US

Serial No.09/302,471

: Group Art Unit 1755

Filed April 30, 1999

: Examiner D. Brunsman

SILICA-BASED COATING FILM ON
SUBSTRATE AND COATING SOLUTION
THEREFOR

THE COMMISSIONER IS AUTHORIZED
TO CHARGE ANY DEFICIENCY IN THE
FEES FOR THIS PAPER TO DEPOSIT
ACCOUNT NO. 23-0975

SUBSTITUTE BRIEF

Assistant Commissioner for Patents
Washington, DC 20231

Sir:

This is an appeal from the Final Rejection of claims 7 to 15.

This Brief replaces the one filed on July 31, 2002 and is responsive to points set forth in the Notification dated September 6, 2002.

1. REAL PARTY IN INTEREST.

The real party in interest is Tokyo Ohka Kogyo Co., Ltd. of Japan.

2. RELATED APPEALS AND INTERFERENCES.

There are no related appeals or interferences.

3. STATUS OF CLAIMS.

The claims are 7 to 15, all of the claims in this application.

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4. STATUS OF AMENDMENTS.

There are no amendments filed subsequent to final rejection.

5. SUMMARY OF THE INVENTION.

The present invention, as defined in claims 7 to 12, relates to a method for preparing a coating solution containing an organopolysiloxane and claims 13 to 15 relate to a method for the formation of a silica-based coating film on a substrate which comprises coating the substrate with a coating solution of organopolysiloxane.

The method of producing the organopolysiloxane solution of claim 7 starts with dissolving a polyalkoxysilane in an alcohol solvent to give a solution which is then mixed with a basic compound and water to effect hydrolysis and condensation of the polyalkoxysilane so as to give an organopolysiloxane.

Then, the first organic (alcohol) solvent is replaced with a second organic solvent which is an aprotic polar organic solvent to obtain the desired concentration (5 to 25 % by wt.), calculated as SiO_2 of organopolysiloxane therein.

In the coating method of claims 13 to 15, organopolysiloxane solution, after being coated on a substrate is heated to form a dried coating layer which is then baked to provide a silica-based coating film on the surface of the substrate.

In order to prepare the presently claimed coating solution, the polyalkoxysilane compound is dissolved in the first organic (alcohol) solvent to give a solution having a concentration of from 1 to 5 % by weight calculated as SiO_2 . The reason for such concentration of silane is to prevent gelation. See page 6, first full paragraph of the present specification.

The hydrolysis/condensation of the polyalkoxysilane proceeds by the addition of water and a basic compound as a catalyst.

The concentration of the catalyst controls the rate of the hydrolysis reaction, as pointed out on page 7, last full paragraph of the present specification and the preferred amount of catalyst is recited in claim 10.

If the reaction proceeds too rapidly, undesirable gelation may result.

Then, the alcohol solvent is replaced with an aprotic organic solvent. The reason for the solvent replacement is, as set forth in the paragraph bridging pages 8 and 9 of the specification, that the storage stability of the solution can be greatly improved.

Further, as pointed out in the second full paragraph on page 9 of the present specification, concentration of the hydrolysis-condensation product of organopolysiloxane is 5 to 25 % (claim 7(c)) calculated as SiO₂ which, in spite of being substantially higher than the solution in the first organic solvent, i.e. the alcohol solvent, is free from gelation.

6. ISSUES.

The issues on appeal are the propriety of the rejection of claims 7 and 10 to 15 under 35 USC 102(b) as anticipated by Takei et al. and the rejection of claims 8 and 9 as unpatentable under 35 USC 103(a) as being unpatentable over Takei et al. further in view of Tomikawa et al.

7. GROUPING OF CLAIMS.

Claims 7 to 9 and 12 stand or fall together.

Claim 10 does not stand or fall with the remaining claims.

Claim 11 does not stand or fall with the remaining claims.

Claims 13 to 15 stand or fall together.

8. ARGUMENT.

The rejection of claims 7 and 10 to 15 as anticipated by Takei et al. (JP Kokai 9-208237) is respectfully traversed for the following reasons:

1. The final rejection asserts that Takei discloses a method for forming a coating composition. However, Takei is directed to a method for the preparation of silica glass and not a coating composition. Example 1 of Takei in fact discloses that 4.5 kg of a silica sol solution were taken portionwise in four 480 mm square vats and converted therein into silica gel, probably in the form of blocks or boards. No coating composition was prepared in Example 1 as well as in Example 2 and Comparative Example 1.

2. The silicon compound dissolved in a solvent in step (a) of present claims 7 and 10 to 12 is a polyalkoxysilane such as TMOS (tetramethoxysilane). On the other hand, the starting silane compound dissolved in a solvent in the first approach of Takei's method is not such an alkoxysilane compound *per se* but rather a partial polycondensation product of a silicon alkoxide.

3. The amount of water to be added to the polyalkoxy silane solution is 2 to 20 moles per mole of the silane (present claim 11). Assuming that the silane is tetramethoxysilane (TMOS) having a molecular weight of 152, this molar proportion corresponds to the ratio of 36/152 to 360/152 by weight. On the other hand, Takei teaches that the concentration of water in the starting mixture must not exceed 1% by weight, clearly teaching away from a higher concentration of water (see section [0006] of the English translation of Takei of record).

Thus, claim 11 is even more remote from Takei than claims 7 to 9.

4. In the starting solution in step (a) of present claims 7 and 10 to 12, the alcohol solvent in the solution is subsequently replaced with an aprotic polar solvent in step (c) while Takei et al. gives not the slightest hint of such a step of solvent replacement.

5. In present claims 7 and 10 to 12, the solvent in which the starting silicon compound is dissolved is an alcohol solvent while, in Example 1 of Takei, the starting silicon compound is dissolved in a mixture of methanol, DMF and tetrahydrofurfuryl alcohol (not THF, i.e. tetrahydrofuran).

6. There is no teaching of the amount of the basic compound as a catalyst as recited in claim 10 nor any appreciation of the need to control the amount of basic compound, e.g. as set forth on page 7, first full paragraph of the present specification, i.e. so that the reaction proceeds at a moderate rate.

If the reaction fails to proceed at a moderate rate, there is a risk of gelation.

Thus, claim 10 is even more remote from Takei than claims 7 to 9.

Claims 13 to 15 also cannot be anticipated by Takei et al.

In this regard, these claims recite a coating method and no coating method is specifically disclosed by Takei.

Moreover, claims 13 to 15 require the formation of a coating layer from an organopolysiloxane dissolved in an organic solvent in the presence of a basic compound. In contrast, the reference relates to the manufacture of silica glass by the sol-gel process and is unconcerned with producing coating layers.

With regard to the rejection of claims 8 and 9 as being unpatentable over Takei et al. above in view of Tomikawa et al. (JP Kokai 6-83063), it is evident from the full English translation of Tomikawa of record that there is nothing in Tomikawa which overcomes the above-discussed deficiencies of Takei. For example Tomikawa makes no mention of solvent replacement.

CONCLUSION

For the foregoing reasons, it is apparent that the rejections on prior art are untenable.

APPENDIX.

A copy of the claims on appeal is set forth in an Appendix immediately following the signature.

This brief is submitted in triplicate and the requisite fee of \$320.00 has already been paid.

Respectfully submitted,

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APPENDIX - CLAIMS ON APPEAL

7. A method for the preparation of a coating solution which comprises the steps of:

- dissolving a polyalkoxy silane compound represented by the general formula



in which R^1 is a hydrogen atom or a monovalent hydrocarbon group, R^2 is an alkyl group and the subscript n is 2, 3 or 4, in a first organic solvent, which is an alcohol solvent, to give a solution, in a concentration in the range from 1 to 5% by weight calculated as SiO_2 ;

- admixing the solution with a basic compound and water to effect hydrolysis of the polyalkoxy silane compound so as to give an organopolysiloxane as a hydrolysis-condensation product thereof; and

- replacing the first organic solvent in the solution with a second organic solvent, which is an aprotic polar organic solvent, in such an amount that the concentration of the organopolysiloxane in the solution is in the range from 5 to 25% by weight calculated as SiO_2 .

8. The method for the preparation of a coating solution as claimed in claim 7 in which the basic compound is ammonia or an amine compound.

9. The method for the preparation of a coating solution as claimed in claim 8 in which the basic compound is ammonia.

10. The method for the preparation of a coating solution as claimed in claim 7 in which the amount of the basic compound is in the range from 10^{-1} to 10^{-5} mole per mole of the polyalkoxy silane compound.

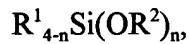
11. The method for the preparation of a coating solution as claimed in claim 7 in which the amount of water is in the range from 2.0 to 20 moles per mole of the polyalkoxy silane compound.

12. The method for the preparation of a coating solution as claimed in claim 7 in which the aprotic polar organic solvent is selected from the group consisting of N-methyl pyrrolidone, dimethylformamide and dimethylacetamide.

13. A method for the formation of a silica-based coating film on the surface of a substrate which comprises the steps of:

(1) coating the surface of a substrate with a coating solution comprising, as a uniform solution,

(A) an organic solvent, and
(B) an organopolysiloxane, which is a hydrolysis-condensation product of a polyalkoxy silane compound represented by the general formula



in which R^1 is a hydrogen atom or a monovalent hydrocarbon group, R^2 is an alkyl group and the subscript n is 2, 3 or 4, dissolved in the organic solvent in the presence of a basic compound, to form a coating layer;

(2) drying the coating layer by heating to form a dried coating layer;
(3) subjecting the dried coating layer to a baking treatment at a temperature of 350°C or higher.

14. The method for the formation of a silica-based coating film on the surface of a substrate as claimed in claim 13 in which the temperature of the baking treatment in step (3) is in the range from 350 to 800°C .

15. The method for the formation of a silica-based coating film on the surface of a substrate as claimed in claim 13 in which the temperature of heating for drying in step (2) is in the range from 80 to 300°C .